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Future Dental Journal of Egypt

Volume 5 | Issue 1 Article 4

2019

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Hassan Shohdy, Engy I.; Nossair, Shereen A.; and Hamdy, Amina M. (2019) "The Effect of Artificial Aging and Surface treatments on Microtensile Bond Strength of Resin Cement to Hybrid Ceramics," *Future Dental Journal of Egypt*: Vol. 5 : Iss. 1 , Article 4.
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The Effect of Artificial Aging and Surface treatments on Microtensile Bond Strength of Resin Cement to Hybrid Ceramics

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ARTICLE INFO

Keywords:

Zirconia-reinforced lithium silicate

Hybrid resin-nanoceramic

Thermocycling

ABSTRACT

Purpose: To assess the effect of aging and various surface treatments on the microtensile bond strength of resin cement to two hybrid esthetic materials.
Materials and Methods: Zirconia-reinforced lithium silicate *(Celtra® Duo, Dentsply Sirona, United States)* and hybrid resin nanoceramic *(Cerasmart ™, GC Corporation, Japan)* blocks were cut into plates of 3 mm thickness. Each ceramic material was surface treated either with hydrofluoric acid etching and silane or with sandblasting and silane. A dual-cured adhesive resin cement *(Panavia ™ V5, Kuraray, U.S.A.)* was utilized to bond the composite resin plates to the surface treated ceramic plates. The ceramic-composite resin blocks were then stored for 24 hours in distilled water. Each block was cut into microbeams and 20 specimens from each subgroup were tested directly after storage while the other half after 5000 thermocycles. Microtensile bond strength test was performed until bonding failure. Three-way ANOVA and Bonferroni's post-hoc test were applied to analyze the data ($P \leq 0.05$).
Results: Both ceramics showed a statistically significant decrease in bond strength after aging. Before aging, zirconia-reinforced lithium silicate showed no statistically significant difference in microtensile bond strength between the two surface treatments. After aging, hydrofluoric acid etching showed higher statistically significant microtensile bond strength than sandblasting. With hybrid resin nanoceramic, hydrofluoric acid etching showed higher statistically significant microtensile bond strength than sandblasting before and after aging.
Conclusion: For both hybrid ceramic materials, aging had a detrimental effect on the bond strength. Moreover, hydrofluoric acid etching as a surface treatment yielded higher microtensile bond strength than sandblasting.

1. Introduction:

The improvement and abundance of ceramic materials has paced significantly in the past decades due to their biological and esthetic virtues. These properties enable it to perfectly mimic the appearance of natural teeth [1]. The newly introduced nano-ceramic materials claim to provide the benefits of simple usage as in composite resin materials, with the strength and surface finish of ceramics [2]. The newest generation of lithium disilicate ceramics (LDC) is zirconia-reinforced lithium silicate *(Celtra® Duo, Dentsply Sirona, United States)* which is composed of 10% zirconia dissolved in lithium silicate glass matrix producing silicate crystals which are 4 times smaller, attributing a high glass content and superior translucency than conventional LDC [3]. It combines the favorable material features of zirconia (ZrO_2) and glass

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Published by Arab Journals Platform, 2019

ceramics in which zirconia particles work as reinforcing fillers with the intention of improving fracture resistance through crack interruption [4]. On the other hand, the manufacturers' rationale to evolve resin-matrix ceramic materials was to obtain a material that has a modulus of elasticity comparable to that of dentin when compared to conventional ceramics, and easier to adjust and mill than glass-matrix or polycrystalline ceramics. It also facilitates both alteration or repair via composite resin [5].

The latest innovated brand of resin–matrix ceramics is called hybrid resin nanoceramic *(Cerasmart, GC Corporation, Japan)*. It is composed of fillers (71 wt %) in the form of barium glass (300 nm), silica (20 nm) and monomer in the form of 2, 2-Bis (4-methacryloxypolyethoxyphenyl) propane (Bis-MEPP), urethane dimethacrylate (UDMA), as well as dimethacrylate (DMA). It has a flexural modulus of 7.5 GPa and flexural strength of 231 MPa [6].

Adhesive bonding systems are used in the dental field not only to promote the retention but also to attain superior esthetic outcomes and ensure high ceramic strength. Bonded all-ceramic restorations demonstrate an improved fracture resistance than traditionally cemented restorations [7]. This emerges as resin cements are elastic in addition to their tendency to deform under stress, conducting a higher fracture resistance [8].

Little is known about the proper surface treatment of zirconia-reinforced lithium silicate ceramics and hybrid resin nanoceramics and the effect of artificial aging on them. The purpose of this study was to assess the effect of aging on the microtensile bond strength (μTBS) of resin cement to the two CAD/CAM hybrid materials.

Materials & Methods:

Zirconia-reinforced lithium silicate (ZLS) *(Celtra® Duo)* and hybrid resin nanoceramic (RNC) *(Cerasmart ™)* blocks were cut into plates of 3 mm thickness using water cooled diamond blade with a low speed cutting saw (*Isomet 4000, Buehler, Lakebluff, USA)*. For each group, four plates were used (4 plates for ZLS and 4 plates for RNC). The plate dimensions of both ZLS and RNC materials are (12 X 14 X 3 mm). The ZLS plates were placed in the ceramic furnace (*Ivoclar P300, Ivoclar Vivadent Inc. Schaan, Liechtenstein)* for further maturation (The cycle specifications: Pre-drying & Preheating 4:00 min, Start temperature: 500°C, Heating rate: 55°C/min, Final temperature: 820˚C). Plates from each CAD/CAM restorative material were wet ground on only one surface using 400, 600- grit silicon carbide (*SiC paper, 3M ™ Wet or Dry Polishing Paper, 3M Espe, St Paul, Minnesota, USA*) which was followed by 5 minutes ultrasonic cleaning in distilled water.

Plates from both CAD/CAM restorative materials were grouped according to the surface treatment applied. Hydrofluoric acid etching (HF) (*IPS® ceramic etching gel, < 5%, Ivoclar Vivadent AG, Schaan, Liechtenstein*) was done for 30 seconds in case of ZLS plates and 60 seconds in case of RNC plates, then washed for 180 seconds with air-water spray and finally it was dried with oil-water free compressed air. On the other hand, a custom-made metal frame was fabricated to hold the plates during sandblasting (SB) which was performed using 50 μm Al_2O_3 particles (*Oxido-de Aluminio, Bio-art, Brazil*) [10 sec, 1 cm and 2 bar pressure in a circular direction]. The plates were then ultrasonically cleaned for 5 minutes in distilled water, then sprayed with alcohol and air dried with oil-water free compressed air.

Scanning Electron Microscope (SEM) was done to investigate the surface topograghy of ZLS and RNC plates. Scanning was done using 10000X magnification for both the untreated and treated surfaces using either HF or SB. The plates of the ceramic materials were fixed to a metal disc holder inside the specimen chamber. The

specimens were then imaged in a low vacuum mode which eliminates the need for coating.

A Teflon mold of dimensions similar to the cut ceramic plates was fabricated to prepare the composite plates. Flowable dual cured core build-up composite **(***Core-Flo ™ DC Lite, Bisco, USA)* was injected inside the mold using an automix tip until the mold was full. Then microscopic glass slide was placed to compress the last increment. Composite was then light cured for 20 seconds via a high intensity (1200 mw/ cm^2) LED unit *(Bluephase, Ivoclar Vivadent Inc., Schaan, Liechtenstein)* from each surface to ensure optimal polymerization. The composite plate was taken off the teflon mold carefully and further light cured for 20 seconds on the areas that were previously in contact with the mold. The plates bonding surfaces were ground by 600-grit SiC paper. Ethanol (70%) was used to wipe and cleanse the surface.

Ceramic Primer (*Clearfil ™ ceramic primer plus, Kuraray, USA)* was applied on all the treated surfaces via a micro-brush in uninterrupted strokes, and air dried with oil-water free compressed air after 1 minute. Another teflon mold (12 X 14 X 6.1 mm) was fabricated where the treated ceramic plate was placed inside it, followed by the generous application of adhesive resin cement *(Panavia ™ V5, Kuraray, USA)* on the ceramic plate, followed by the placement of the composite plate above the resin cement, and finally a fixed load (500 gm) was positioned and was light cured using *Blue-phase* LED unit from each surface to ensure optimal polymerization. The ceramic- composite resin block was then taken off the teflon mold, further light curing was done for 20 seconds, then kept in distilled water for 24 hours before the following step.

The ceramic-composite resin blocks were fixed on epoxy resin cylinders, and each was vertically sectioned into serial slabs using water cooled diamond blade with a low speed cutting saw (Fig. 1). It was then rotated 90° to make additional vertical cuts (Fig. 2), so that the ceramic-composite resin block has perpendicular cuts (Fig. 3) to obtain a thin long micro-beam with the following dimensions (1 mm × 1 mm × 6.1 mm) with an overall cross-sectional area of approximately 1 mm^2. The microbeams were further measured with a digital caliper before bond strength testing as shown in (Fig.4).

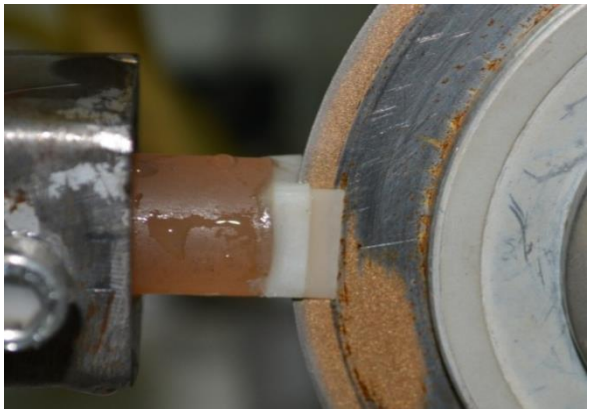

Fig. 1: Ceramic- composite resin block vertically sectioned into serial slabs

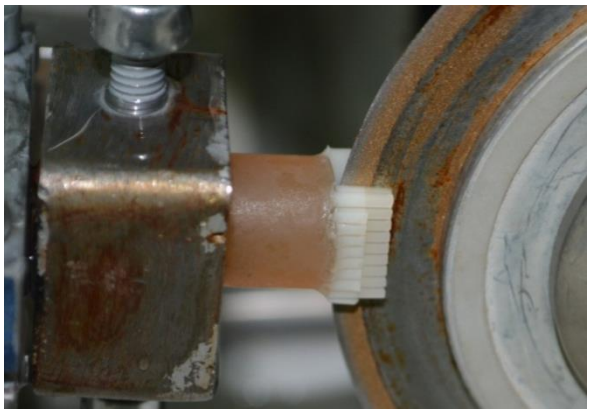

Fig. 2: The ceramic- composite resin block rotated 90◦ to make additional vertical cuts

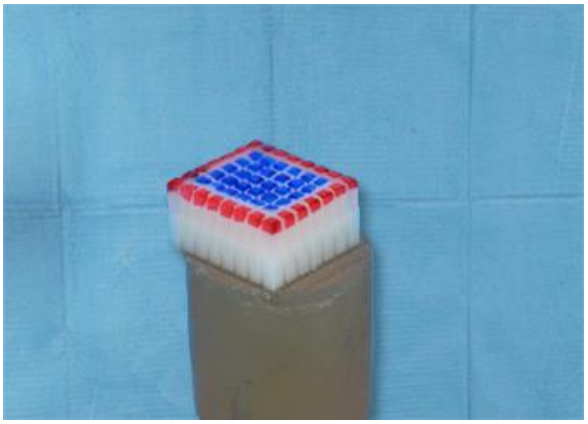

Fig. 3: The ceramic-composite resin block with perpendicular cuts, Red color represents the excluded micro-beams while the blue color represents the used micro-beams

Fig. 4: A micro-beam of 1mm^2 cross-sectional area

Twenty micro-beams were taken out from each block excluding the use of the peripheral slices to avoid the use of slices with an excess or a deficient amount of cement at the interface. Half of the micro-beams from each subgroup (n=20) was tested after 24 hours storage in distilled water, and the other half (n=20) was tested after being subjected to 5000 cycles inside the thermocycling machine (*Thermocycler Willytec THE- 1100, SD Mechatronik GmbH, Feldkirchen-Westerham, Germany*) between (5 - 55 ˚C) with dwell time (20 sec) in each bath.

Then Geraldeli's jig [9] was used to hold the microbeams onto the universal testing machine *(Instron 3345, Instron, Norwood, Massachusetts, USA)*. Each microbeam was placed in the central groove of the jig, glued in place by its ends using cyanoacrylate-based glue and left for a while to harden. After that, all the specimens were subjected to a tensile load (load cell= 500 N, cross-head speed = 0.5 mm/min) until bonding failure of the specimen occurred to gather data about the μTBS before and after aging. The load at failure (N) and the surface area (mm^2) for each specimen was utilized to calculate the μTBS in MegaPascal (MPa) by *Bluehill Lite software (Instron, Norwood, Massachusetts, USA).* Finally, data were statistically analysed.

Debonded microbeams were carefully removed from the jig with a scalpel and stored in their corresponding labelled plastic cones until examination of failure mode. After that the fractured surfaces of each debonded microbeam was examined under a stereomicroscope *(Nikon MA 100, Nikon, Japan)* with a 50X magnification to determine the mode of failure. The failure modes were categorized as follows: adhesive failure (surface of the CAD/CAM material was visible); mixed failure in the CAD/CAM material and cement surfaces (resin cement was partially visible in certain areas); or cohesive failure within the cement layer (almost all the fracture surface was covered with cement).

3. Statistical Analysis:

In this study, three-way ANOVA was used to study the effect of ceramic type, surface treatment, aging and their interaction on mean μTBS. Bonferroni's post-hoc test was used for pair-wise comparisons when ANOVA test is significant. The significance level was set at $P \leq 0.05$. Statistical analysis was done with *IBM* (IBM Corporation, NY, USA) SPSS (SPSS, Inc., an IBM Company) Statistics Version 20 for Windows.

4. Results:

Three-way ANOVA showed significant difference between the ceramic type, surface treatment and aging but on the other hand it showed no significant difference on their interactions as shown in table (1).

Table 1: Three-way ANOVA results for the effect of different variables on mean micro-tensile bond strength

Source of variation	Type III Sum of Squares	Df	Mean Square	F-value	P-value
Ceramic type	547.0	1	547.0	29.6	<0.001*
Surface treatment	960.0	1	960.0	51.9	<0.001*
Aging	6147.4	1	6147.4	332.1	<0.001*
Ceramic type X Surface treatment X Aging interaction	43.7	1	43.7	2.4	0.129

*df: degrees of freedom = (n-1), *: Significant at P ≤ 0.05*

Statistical analysis showed that there was a statistically significant decrease in mean μTBS after aging in table (2). Using either ZLS or RNC whether with HF or SB, there was a statistically significant decrease in mean μTBS after aging (Fig. 5). In case of ZLS with HF, μTBS was significantly higher before aging (28.6 ± 4.0 MPa) than after aging (19.7 ± 4.9 MPa), and with SB it was also significantly higher before aging (29.4 ± 5.4 MPa) than after aging (12.2 ± 2.9 MPa). In case of RNC, the results were significantly higher with HF before aging (43.4 ± 3.4 MPa) than after aging (22.5 ± 3.1 MPa) as well as SB in which the μTBS was significantly higher before aging (34.1± 6.7 MPa) than after aging (10.8 ± 2.5 MPa).

Table 2: The mean, standard deviation (SD) values and results before and after aging of each ceramic type with each surface treatment

Ceramic type	Surface ttt	Before aging		After aging		*P*-value
		Mean	SD	Mean	SD	
Celtra Duo	HF	28.6	4.0	19.7	4.9	<0.001*
	SB	29.4	5.4	12.2	2.9	<0.001*
Cerasmart	HF	43.4	3.4	22.5	3.1	<0.001*
	SB	34.1	6.7	10.8	2.5	<0.001*

**: Significant at P ≤ 0.05*

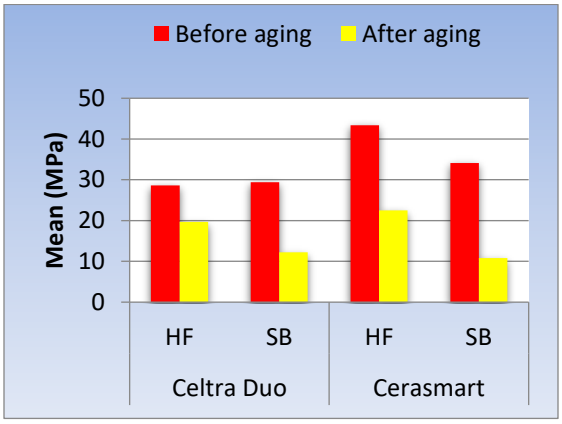

Fig. 5: Bar chart representing mean micro-tensile bond strength before and after aging of each ceramic type with each surface treatment

Regarding the surface treatment, HF showed statistically significant higher mean μTBS than SB as shown in (Fig. 6). Before aging, ZLS showed no statistically significant difference between mean μTBS values of HF (28.6 ± 4.0 MPa) and SB (29.4 ± 5.4 MPa) as shown in table (3). After aging, HF acid etching (19.7 ± 4.9 MPa) showed statistically significant higher mean μTBS than SB (12.2 ± 2.9 MPa). Using

RNC whether before or after aging, HF showed statistically significant higher mean μTBS than SB. Before aging, HF was (43.4 ± 3.4 MPa) while SB was (34.1± 6.7 MPa). And after aging, HF was (22.5 ± 3.1 MPa) while SB was (10.8 ± 2.5 MPa)

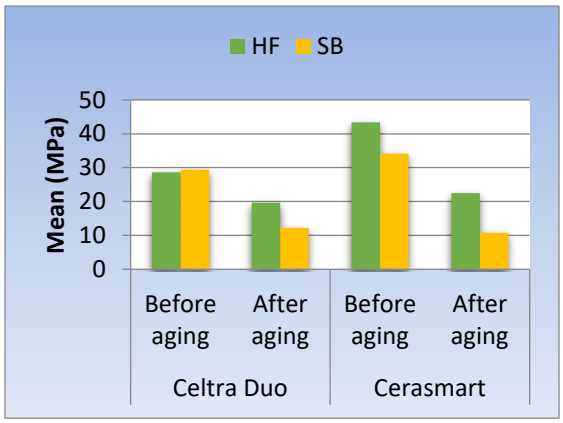

Fig. 6: Bar chart representing mean micro-tensile bond strength of the two surface treatments with each ceramic type before and after aging

Table 3:The mean, standard deviation (SD) values and results of the two surface treatments with each ceramic type before or after aging

Ceramic type	Aging	HF		SB		*P*-value
		Mean	SD	Mean	SD	
Celtra Duo	Before	28.6	4.0	29.4	5.4	0.677
	After	19.7	4.9	12.2	2.9	<0.001*
Cerasmart	Before	43.4	3.4	34.1	6.7	<0.001*
	After	22.5	3.1	10.8	2.5	<0.001*

**: Significant at P ≤ 0.05*

SEM results showed that ZLS plate with no surface treatment showed a smooth surface which consists of a glassy phase rich in lithium silicate crystals and characteristic dispersed zirconia fillers with striations due to polishing procedures as in (Fig.7). While after the HF, it showed a honey-comb-like micro-rough porous surface due to HF- acid etching procedure as shown in (Fig. 8). In case of SB, it showed an irregular shaped surface caused by abrasion of the glassy matrix and the reinforcing crystals, and exposure of needle-like crystals due to sandblasting procedure as shown in (Fig. 9).

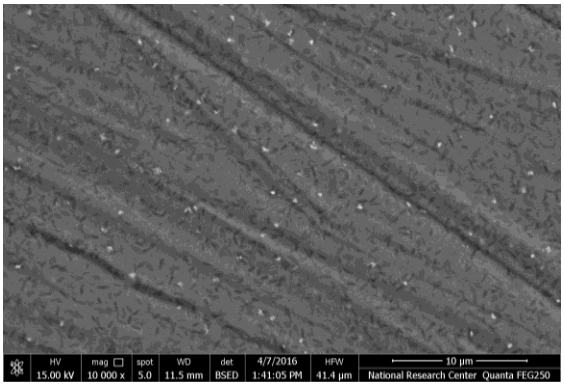

Fig. 7: ZLS plate (10000X) with no surface treatment

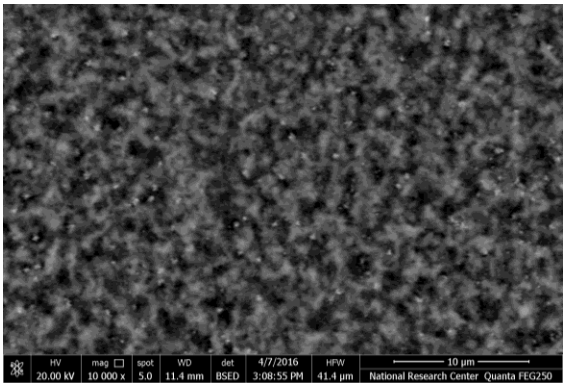

Fig. 8: ZLS plate (10000X) treated with hydrofluoric acid etch

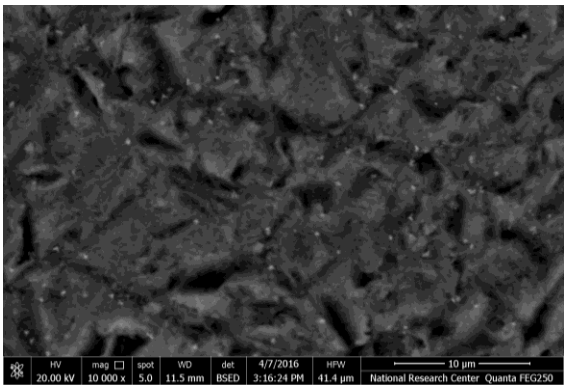

Fig. 9: ZLS plate (10000X) treated with sandblasting

RNC plate with no surface treatment showed a homogenous distribution of the barium glass and silica particles inside the matrix as shown in (Fig.10). While after the HF acid etching it showed the dissolution of the ceramic particles creating micropores as shown in (Fig.11). In case of SB, it showed an irregular surface resulting from the effect of the sandblasting particles on the resin matrix as shown in (Fig.12).

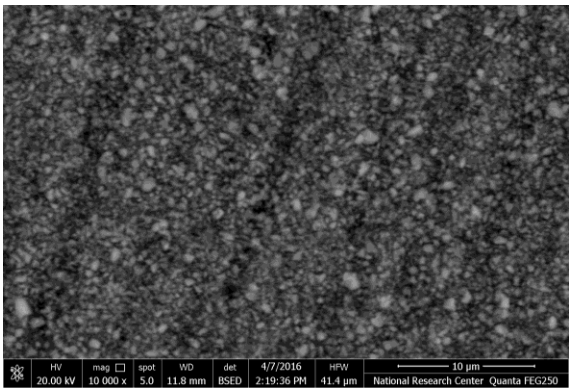

Fig. 10: RNC plate (10000X) with no surface treatment

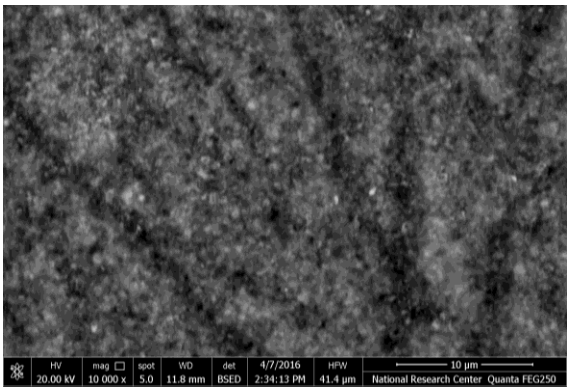

Fig. 11: RNC plate (10000X) treated with hydrofluoric acid etch

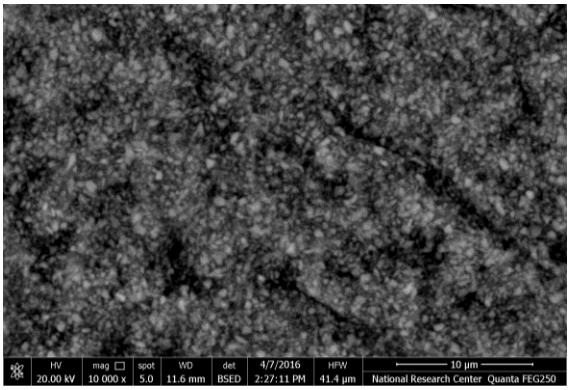

Fig. 12: RNC plate (10000X) treated with sandblasting

Regarding the mode of failure, it was also influenced by the type of surface treatment, ceramic material and aging. The dominant mode of failure for the sandblasted groups is cohesive failure before aging and adhesive failure after aging. While for the hydrofluoric acid etched groups, the dominant mode of failure before and after aging is cohesive failure. These data are summarized in table (4).

Table 4: Mode of Failure of different test groups

Ceramic type	Aging	HF	SB
Celtra Duo	**Before**	50% Cohesive	60% Cohesive
		50% Mixed	40% Mixed
	After	75% Cohesive	75% Adhesive
		25% Mixed	25% Mixed
Cerasmart	**Before**	75% Cohesive	70% Cohesive
		25% Mixed	30% Mixed
	After	80% Cohesive	90% Adhesive
		20 % Mixed	10% Mixed

5. Discussion:

Bonding is usually achieved by 2 synchronized mechanisms; micromechanical retention through either HF acid etching or SB of the ceramic surface and chemical coupling by the application of a silane coupling agent [10]. It improves the surface energy of the ceramics and the wettability of the cement [11]. Various strategies are favored depending on the material's features [12].

The etching of glass ceramics using 4-9.5% HF acid was widely applied and proved to be a very effective etching protocol in creating the micro-mechanical retention required [13]. The surface topography formed after etching relies on the ceramic components and microstructure, the acid concentration and type, and the acid etching duration [14]. On the other hand, sandblasting proved to be an efficient surface treatment to composite prior to cementation [15]. **Gré et al [**16] found that silanization showed higher μTBS compared to groups without silane. Hence in this study, HF acid etching and SB were used as the surface treatments before silanization.

In this study, the hybrid ceramic materials were bonded to composite resin substrates rather than dentin disks to develop a durable bond between the luting system and composite resin allowing the weak connection to be at the CAD/CAM material/cement interface. Otherwise, failures might happen at other sites hiding the surface treatments effects [17]. Additionally, variations in the tooth microstructure could lead to misinterpretation of the findings [18].

Bond strength measurements are among the methods used to assess the efficiency of adhesive systems to predict their performance in the oral cavity. μTBS test is considered as the most accurate bond strength test [19]. So, it was chosen in this study as it allows for appropriate positioning of the specimens, and uniform distribution of the stresses permitting precise evaluation of bond strength values [20].

In this study, HF acid etching showed higher μTBS than SB. Furthermore, the dominant mode of failure for the hydrofluoric acid etched groups, before and after aging is cohesive failure. Previous studies of **Blatz et al** [13] and **Conrad et al [**21] also reported that etching with 4-9.5% HF acid has proved to be an effective surface treatment offering surface roughness for mechanical interlocking for the glass ceramics containing different amounts of glass/silica compositions. This could be justified as HF acid produces micro-porosities on the fitting surface of the restorative materials, increasing the surface area, and enhancing the foundation of mechanical interlocking with resin cements [22].

The lower bond strength observed with SB in comparison to HF could be attributed to the failure of SB to provide true means of undercuts on the abraded surface which was supported by **Kato et al** [23]. Moreover, The dominant mode of failure for the sandblasted groups is cohesive failure before aging and adhesive failure after aging**. Tabatabei et al [**24] also explained that it could be due to either, the surface debris left on surface after treatment or due to the inclusion of air which decreases the available surface area for bonding. Moreover, it was reported that airborne particle abrasion could induce a stress that may be concentrated at the indirect restorative materials/luting agents interface, resulting in sharp angles which may interfere with proper wetting and produce voids at the interface [25].

With regards to the surface treatment, it was found that ZLS showed no statistically significant difference between the μTBS values of the two surface treatments. **Panah et al** [26] and **Yavuz et al [**27] demonstrated that there is no significant difference between a ceramic surface air abraded with Al_2O_3 and one etched with HF. HF acid etching of ZLS in the SEM image showed a characteristic honeycomb-like micro-rough appearance and creating a microporous surface as shown in (Fig.8), by partially dissolving the glass phase a few microns in depth, allowing the lithium silicate crystals to protrude from the glass matrix, leaving behind an active surface rich in silica [28]. It increases the surface area and simplifies the penetration of the resin into the micro-porosities of the etched ceramic surfaces [29].

On the other hand, SB showed abrasion of both the glassy matrix and reinforcing crystals, together with revealing needle-like crystals as shown in (Fig.9). Airborne particle abrasion is a routine step used to remove the reaction layer around pressed LDC, but it can be selectively

used on the fitting surface of CAD/CAM restorations [4].

In case of RNC, HF acid etching showed higher μTBS value than SB. This could be attributed to the hybrid composition of RNC which consists of barium glass (300 nm), silica (20 nm) fillers of 71% by weight and 2,2-Bis (4-methacryloxypolyethoxyphenyl) propane monomer [6]. RNC used in this study has etchable barium-glass particles in addition to a lower hardness as opposed to the zirconia-containing composite CAD/CAM block material used in previous studies [30,31]. Consequently, it is more vulnerable to mechanical roughening and acid etching [32]. HF surface treatment of RNC in the SEM image modified the microstructure by partial dissolution of the ceramic particles creating micropores as shown in (Fig.11). HF is also used to surface treat indirect composites having ceramic fillers before bonding, due to its roughening effect by attacking the exposed ceramic filler in preference [33]. While SEM image of SB showed an irregular surface resulting from the effect of sandblasting particles on the resin matrix as shown in (Fig.12).

Considering the two ceramics chosen in this study, RNC **(**Cerasmart ™) showed higher μTBS than ZLS (Celtra® Duo). This may be due to their different microstructural composition where, RNC consists of silica and etchable barium glass fillers. Thus, there is a predominance of ceramic glass fillers in its composition [34]. **Borges et al** [35] concluded that, the surface treatment efficiency is extremely dependent on the composition of the ceramic substrate rather than the treatment itself. Additionally, polymer-based materials showed comparatively high flexural strength with a low flexural modulus during testing. This combination translates to a higher ability to endure loading by undergoing more elastic deformation prior failure and tend to be more flexible and less brittle [36].

Restorative materials used intra-orally are subjected to a complex humid and wet oral environment which is characterized by natural saliva and its components [37]. It is also subjected to different hot and cold food/ beverages with variable Ph and different chemical compositions. Furthermore, aging and thermocycling are two factors that significantly reduce the bond strength in vitro studies [38]. In thermocycling, samples undergo a number of cycles between (1000 - 100,000) cycles between (5-55) ˚C [39].

Thermocycling decreased the μTBS of both materials because of thermal and hydrolytic degradation and a way to simulate temperature-related failure by repetitive abrupt temperature fluctuations [40]. As it exerts thermal stresses at the interface between the ceramic substrate and the luting agent; due to the alteration in coefficient of thermal expansion and contraction [41].

Water sorption could be a determinable factor in decreasing the bond strength values due the small molecular size along with the high molar concentration of water, it can break through nanometer-size free-volume spaces between polymer chains or cluster around functional groups that have the ability of hydrogen bonding producing polymer plasticization and reducing thermal stability [42]. These results are supported by **Matsumura et al [**43] confirming the explanation that the ceramic-resin bond is vulnerable to hydrolytic degradation in the presence of water.

Thermocycling in relation to the surface treatment showed that after aging HF had a higher μTBS than SB. This could be justified due to the absence of undercuts on the abraded surface by SB, which was supported by **Nagai et al** [44]. Moreover, this could be attributed to the stresses generated at the interface between the ceramic material and the resin cement after aging due to the alteration in coefficient of thermal expansion and contraction between both materials, in addition to the sharp angles on the surface shown in the SEM of the sandblasted specimens, this might be the reason to generate microcracks inside the material leading to debonding. On the other hand, **Menees et al** [45] argued that HF acid etching provides more uniform and better distributed surface changes on LDC.

From what has been previously discussed it can be deduced that the bond strength is multi-factorial and depends on the nature of the ceramic material, surface treatment in addition to the simulated aging factors. Hence, further investigations are essential to evaluate the actual effect of the oral environment on it.

6. Conclusions:

From the foregoing results and within the limitations of this study, it was concluded that:

1. Aging significantly decreased the (μTBS) of resin cement to zirconia-reinforced lithium silicate and hybrid resin nanoceramic materials.
2. The detrimental outcome of aging on μTBS was more obvious with sandblasting.
3. On the level of surface treatments, HF acid etching was more effective than SB as a surface treatment especially with hybrid resin nanoceramics.

7. Limitations of study:

Our study showed some limitations, among which was the effect of aging after sandblasting on the ceramics and the stresses generated at the interface which should be further investigated together with the surface roughness and topography of the ceramic materials. Further in vivo studies are suggested to assess and analyze their clinical performance in the oral environment in daily dental applications to provide reliable recommendations for dental practitioners.

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